2019/10/31 Session TUN-1 C2

19th International Conference on Fusion Reactor Materials(ICFRM-19)

in La Jolla, California, U.S.A.

Thermal Diffusivity of Irradiated Tungsten and Tungsten-Rhenium alloys

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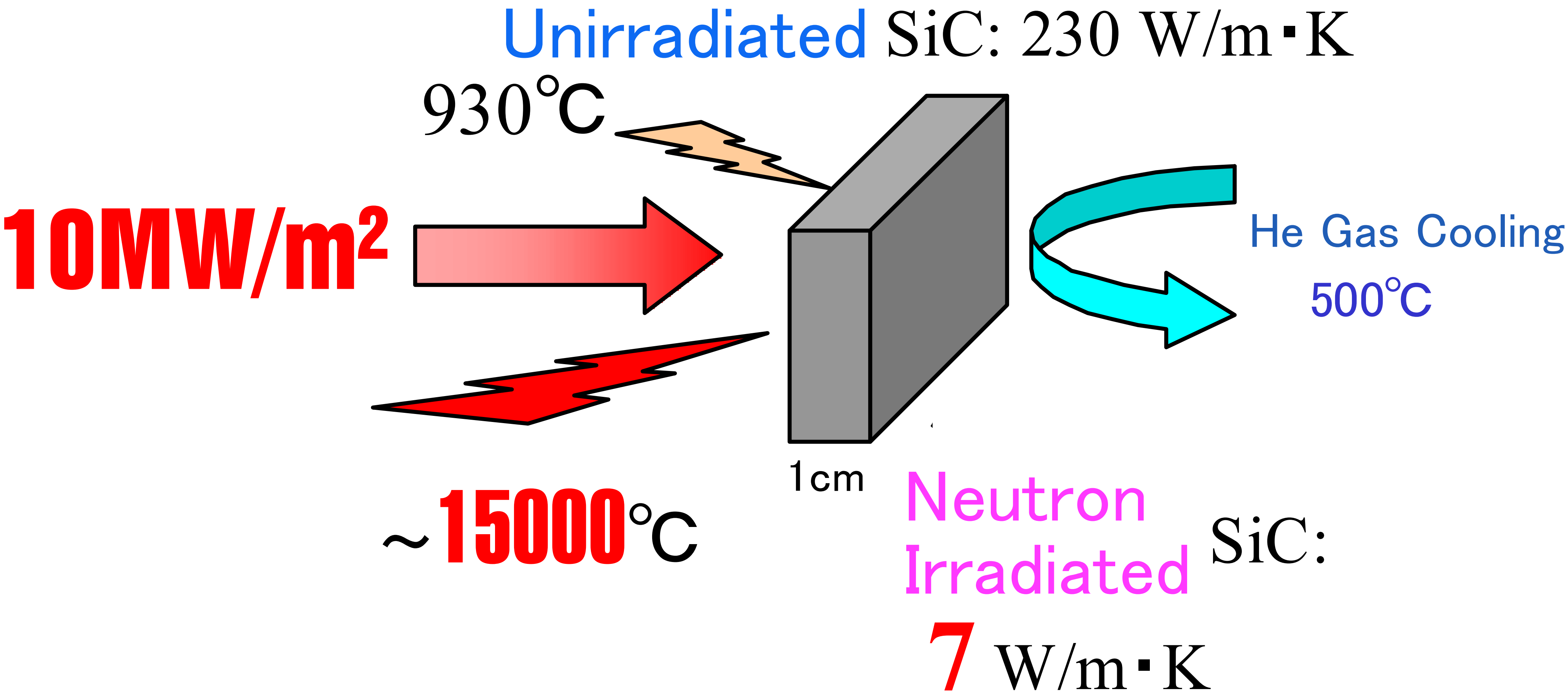
2013-2018 PHENIX

JAPAN-US JOINT PROJECT

Degradation of Thermal Conductivity in Ceramics

Divertor of Fusion Reactor



Thermal diffusivity of Tungsten

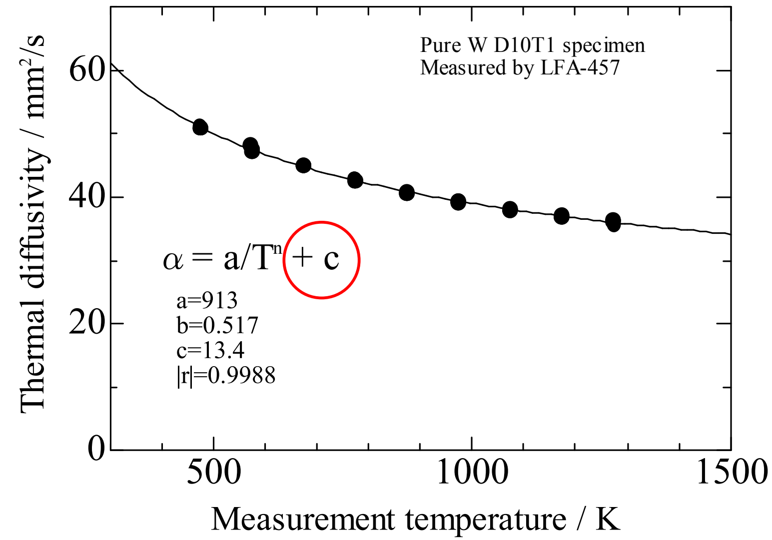
Thermal diffusivity in Metal / Ceramics

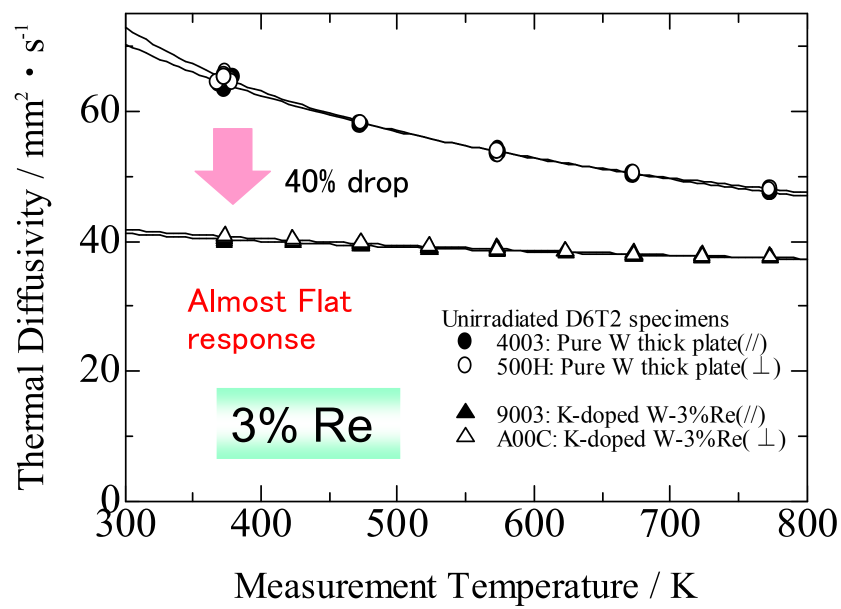
In Tungsten, heat is mainly carried by electron like typical metals, but several part of heat is carried by phonon like ceramics.

In ceramics, high temperature increase phonon-phonon scattering and thermal diffusivity α is described as $\alpha = a/T^n$, where unirradiated specimens show n = 1 and irradiated them show n < 1.

Unirradiated pure tungsten showed n~0.5 and have a constant term as $\alpha = a/T^n + c$.

Furthermore, fusion neutron induces transmutation element such as Rhenium or Osmium that reduce thermal diffusivity drastically.





RB-19J Irradiation in PHENIX Project

In light water fission reactor, most neutrons are moderated to thermal neutrons that gives larger amount of transmutations than the fusion reactor at the same irradiation induced damage.

W → Re, Os

Thermal diffusivity change;

Electrically? Phonon scattering?

In the PHENIX project, irradiation in HFIR have been performed with the **Gadolinium** thermal neutron shield.

Location: RB*

Dose: 0.2~0.7 dpa, 4 cycles

Temperature regions

- 400℃, 406 specimens
- 800℃, 389 specimens
- 1100℃, 359 specimens

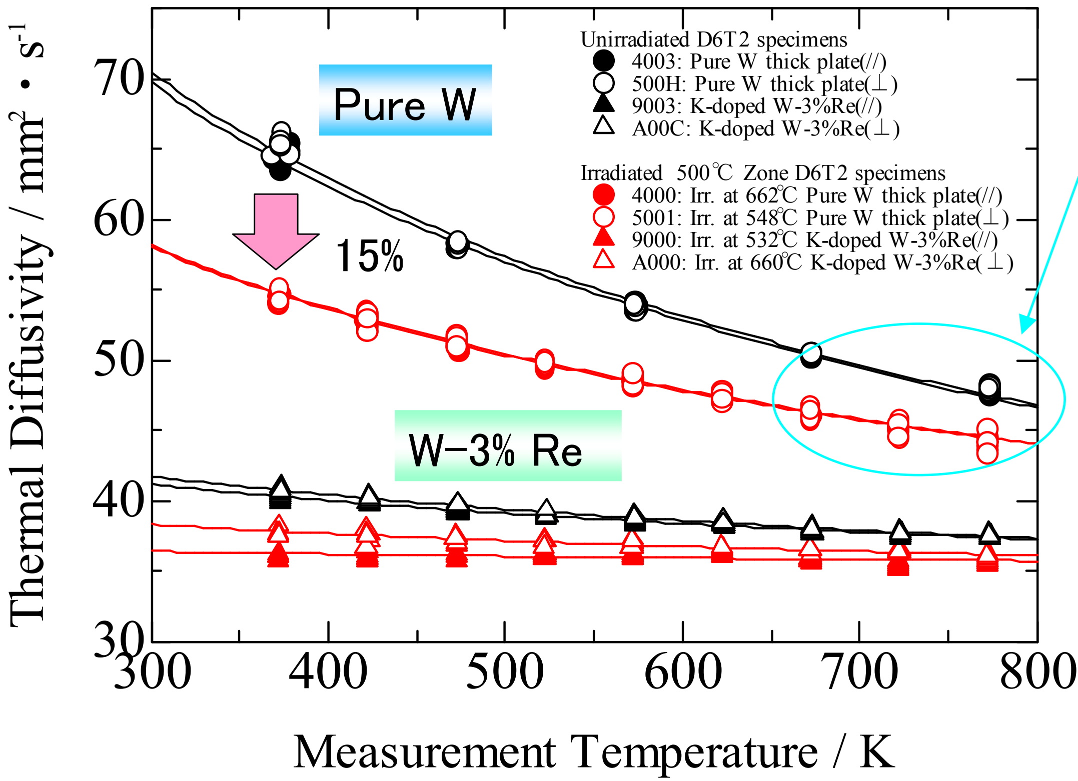
Separate effect of lattice defects from transmutations.

Compare with rapid irradiation without Gd shield or TITAN is important.

Pure W and W-3Re Irradiated specimen

・D6T2 specimens irradiated in 500℃ zone
→ Thermal diffusivity measurement using LFA-457 in LAMDA up to 500℃

Irradiation dose is assumed to be 0.2-0.7dpa for hole 19J capsule, where in the case of 500℃ zone, it may be close to 0.2dpa.



・Irradiated Pure W specimens showed obvious degradation in thermal diffusivity, while they were higher than that of unirradiated K-doped W-3%Re specimens.

・Furthermore, at elevated temperature thermal diffusivity of irradiated pure W were getting close to that of unirradiated.

・It showed that the **transmutation effect was relatively limited**, and only phonon scattering were increased.

・Irradiation effect on K-doped W-3%Re specimens were quite limited. Irradiated specimens showed almost same thermal diffusivity with increasing temperature.

・The difference in grain orientation was not observed. Irradiated K-doped W-3%Re specimens showed a little difference, but it may be arisen from irradiation condition (130℃ different).

Annealing measurement procedure

Specimen irradiated at 500℃

RT → 100℃ → 150℃ → 200℃ → .... → 500℃

As-irradiated measurement

Next day

Cool down in LHA-467HT

RT → 100℃ → 150℃ → 200℃ → .... → 600℃ (60shot)

Isochronal annealing up to 600℃ and also isothermal annealing at 600℃

Next day

Cool down in LHA-467HT

RT → 100℃ → 150℃ → 200℃ → .... → 700℃ (60shot)

Isochronal annealing up to 700℃ and also isothermal annealing at 700℃

Next day

Cool down in LHA-467HT

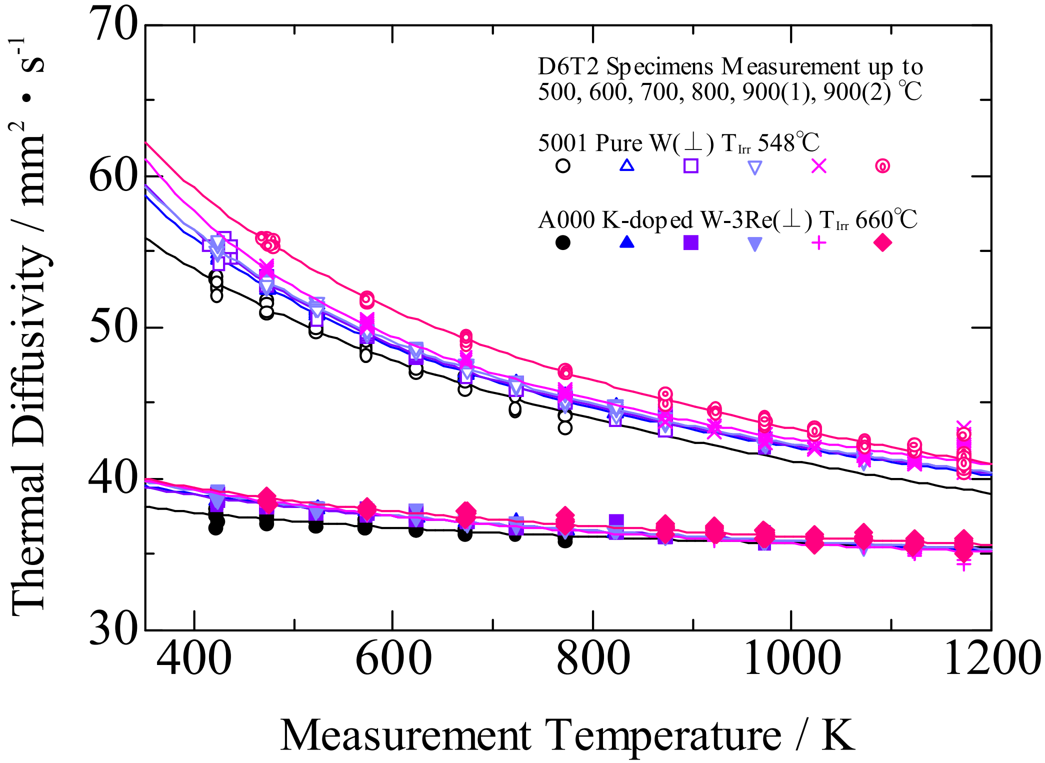
These treatment will be continued up to 1100℃

At each isochronal annealing temperature, dependence on measurement temperature is obtained.

At each step, NO Rad-Work is required.

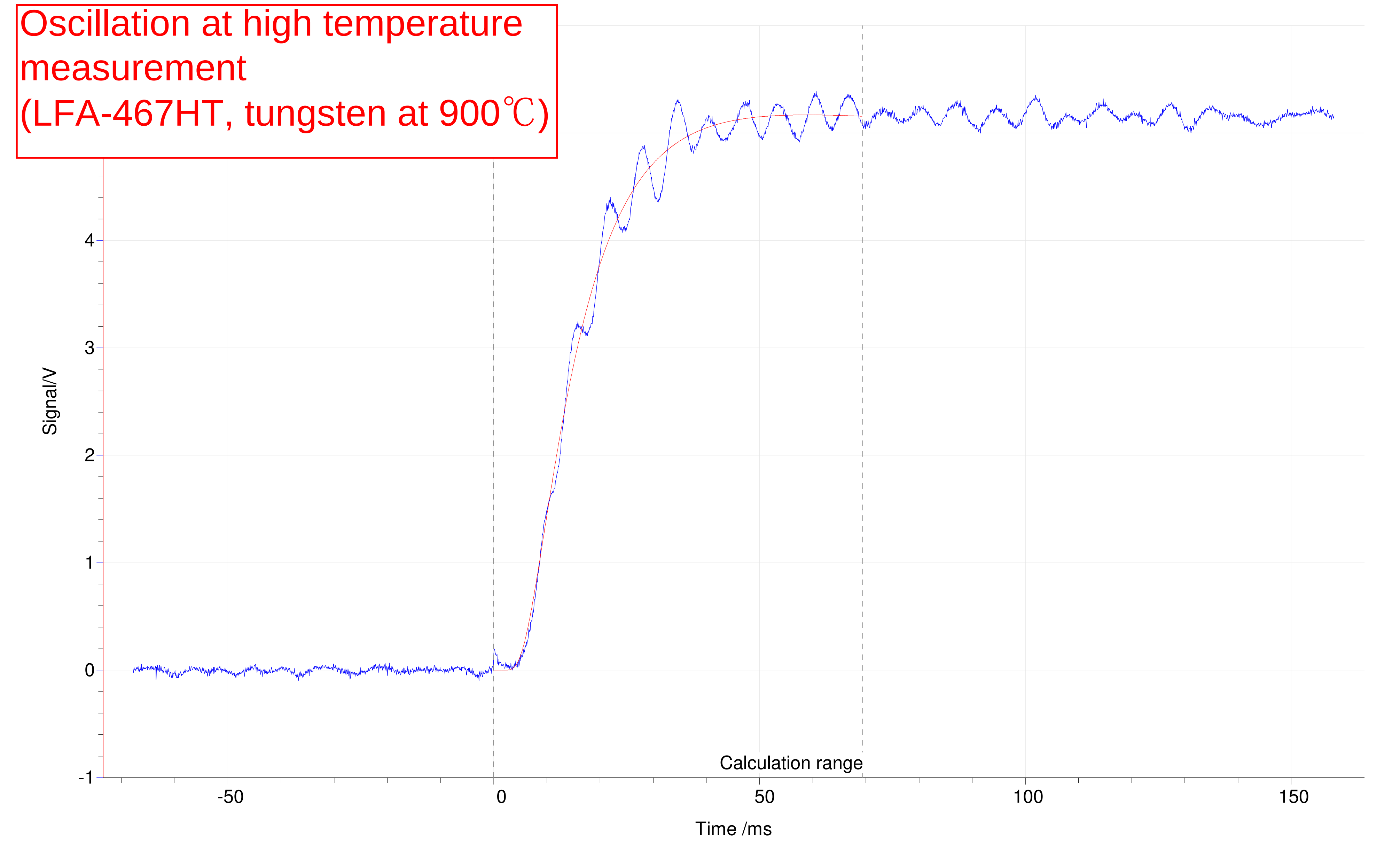
(Liq. N_2 must be supplied)

Annealing effect in irradiated specimens



- Slight annealing recovery was observed at 900℃.
- Now try to continue it up to 1100℃ but a technical problem is not resolved for high temperature measurement using LFA-467HT.
- It looks that only a few point-defects such as vacancies were induced. → Validate using positron annihilation lifetime measurement.
- It is required to compare transmutation effects with Rapid specimens or TITAN specimens.

If only a few point defects that we can ignore are induced, we can forecast a thermal diffusivity after an irradiation with a calculation of transmutation amount (and its distribution).



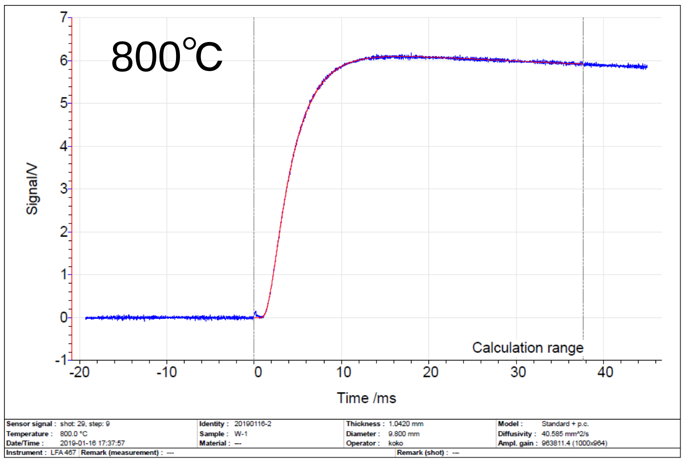
Sensor signal :	shot: 96, step: 12	**Identity :**	Novenver30th-Task1-Annealing-upto90	**Thickness :**	2.0390 mm	**Model :**	Cowan + p.c.
Temperature :	900.0 °C	**Sample :**	5001	**Diameter :**	9.800 mm	**Diffusivity :**	40.942 mm^2/s
Date/Time :	2018-11-30 23:09:42	**Material :**	---	**Operator :**	HW	**Ampl. gain :**	2002562.5 (1000x2003)
Instrument :	LFA 467	**Remark (measurement) :**	---	**Remark (shot) :**	---		

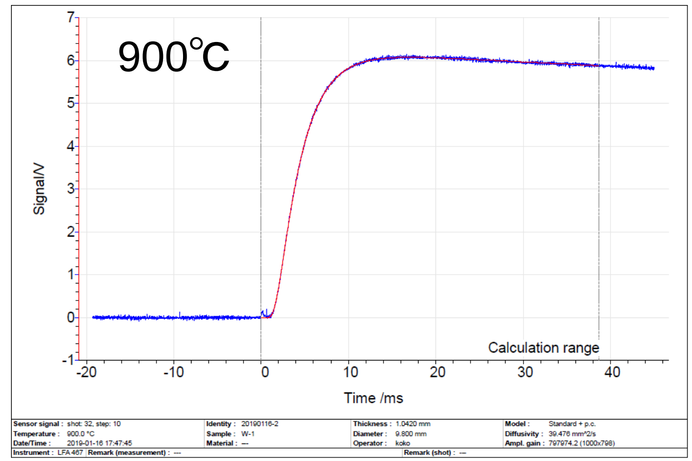
Created with NETZSCH Proteus software

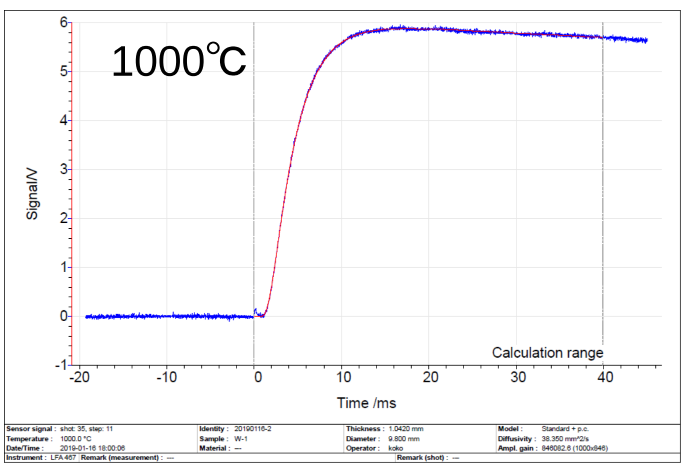
IR signal curve

NETZSCH

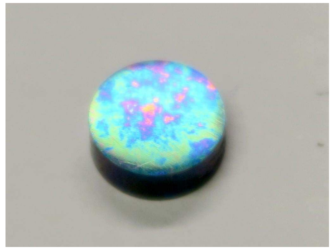
Specimen: Pure W (polished, Graphite spray coating)







Oxidation

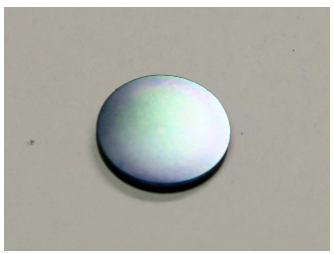



K-doped W-3%Re and Pure W after the measurements up to 500℃ in vacuum via RP using LFA-457.

Pure W up to 800℃

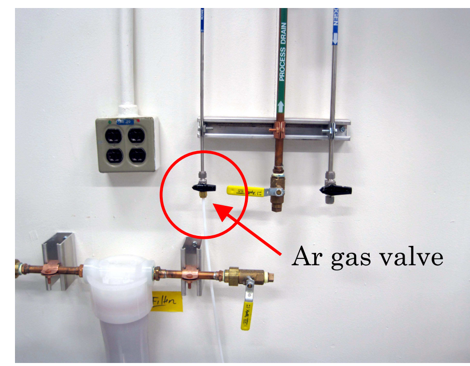
K-doped W-3%Re up to 1100℃

Using LFA-467HT with a Turbo Pump




Pure W (ITER-G) and tungsten side of W/SiC after a measurement up to 500℃ and 800℃ with Ar flow using LFA-457.

Ar leak from a valve inside of LFA-467HT cabinet gives a slight oxidation after a measurement up to 800℃ on Pure W.



To achieve high vacuum, **Ar valve at wall must be closed manually** after purge treatments.

NETZSCH Japan reported there is NO oxidation with Ar flow using LFA-467HT (without turbo pump)

Irradiation effect in tungsten

To separate electron conduction and phonon conduction, electrical resistibility measurement at elevated temperature using specimen with different concentration of Re must be performed.

In addition, positron annihilation lifetime measurement is required to estimate phonon conduction.

Electrical resistibility

(Using miniature specimens) → ORNL

John Echols

Positron annihilation lifetime

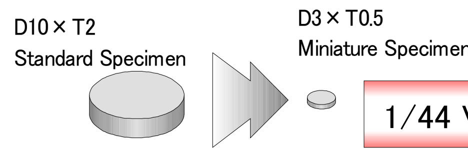
(Using miniature specimens) → Osaka Pref. Univ.

H. Ando

Miniature specimens

Small specimen for thermal diffusivity measurement is strongly required to reduce radio activity and volumetric heating during the neutron irradiation.

PHENIX Project: 2013–2018 6Year
PIE cannot wait the cooling.

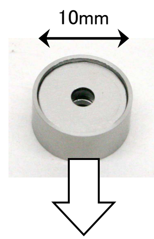


→ TEM Disk Size

1/44 Volume → 1/44 Radio-Activity

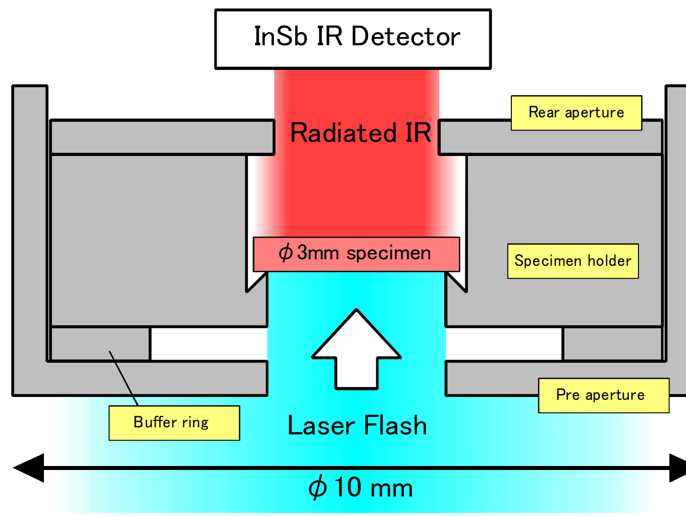
In the PHENIX project, irradiation in HFIR have been performed with the specimen form of Diameter 3mm ×Thickness 0.5mm (D3TH) for thermal diffusivity measurement. Specially manufactured specimen holders enable the measurement of this D3TH miniature specimens.

D3 Specimen holder manufactured by Mo-TEM alloy.



Put In
As D10 Specimen

Netzsch LFA-467HT
Standard Specimen Holder
(For 12.7mm specimen,
with 10mm Conversion ring)



Required thickness of specimen

Pulse width of Laser Flash in LFA-457: T_f = 330 μ s

LFA-467: $T_f \geqq$ 20 μ s

for quality guarantee

ASTM E1461, JIS R1611, Netzsch recommend. → Require $T_f < T_{1/2} / 10$

$\alpha = 0.1388\ t^2 / T_{1/2}$

α: thermal diffusivity, t: thickness, $T_{1/2}$: half time

NETZSCH LFA-457

$T_{1/2} >$ 3.3ms ($T_f \times 10$)

Tungsten (unirradiated), α=66.0mm²/s

→ Thickness $t >$ 1.25mm

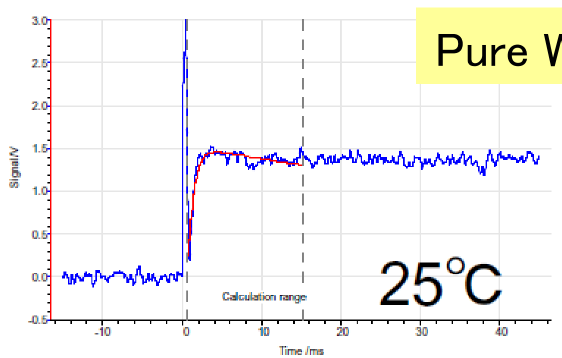
must be larger than

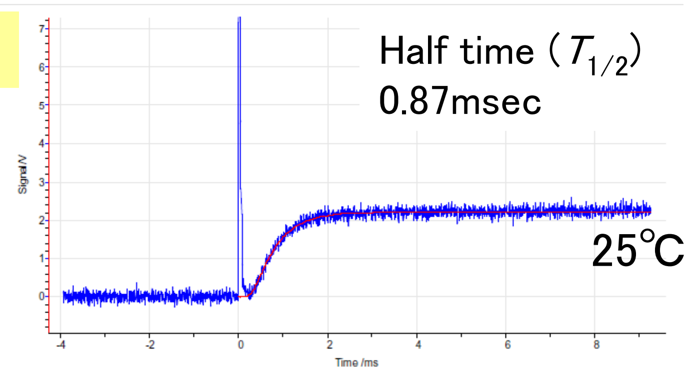
NETZSCH LFA-467

$T_{1/2} >$ 0.2ms → $t >$ 0.31mm

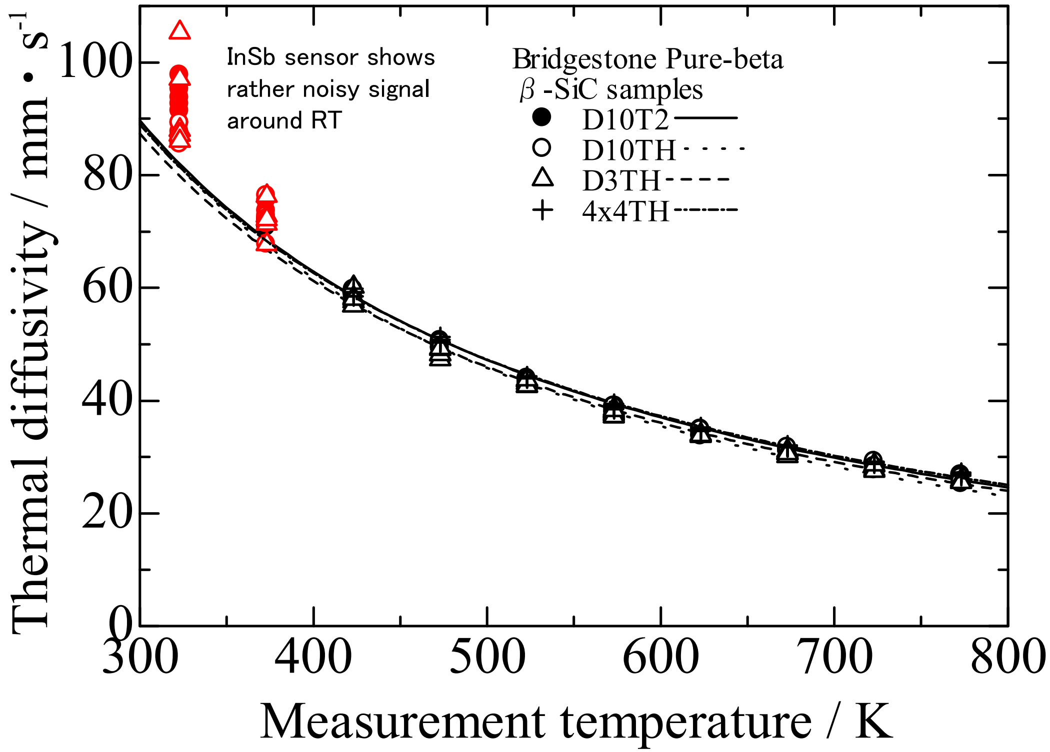
Zoom Optics: IR sensor can focus within D 2.7mm small area

Pure W, D3 t=0.598mm





Validation of measurement using various specimen form using LFA-467HT



Thermal diffusivity of

D10T2, D10TH, D3TH, 4x4TH

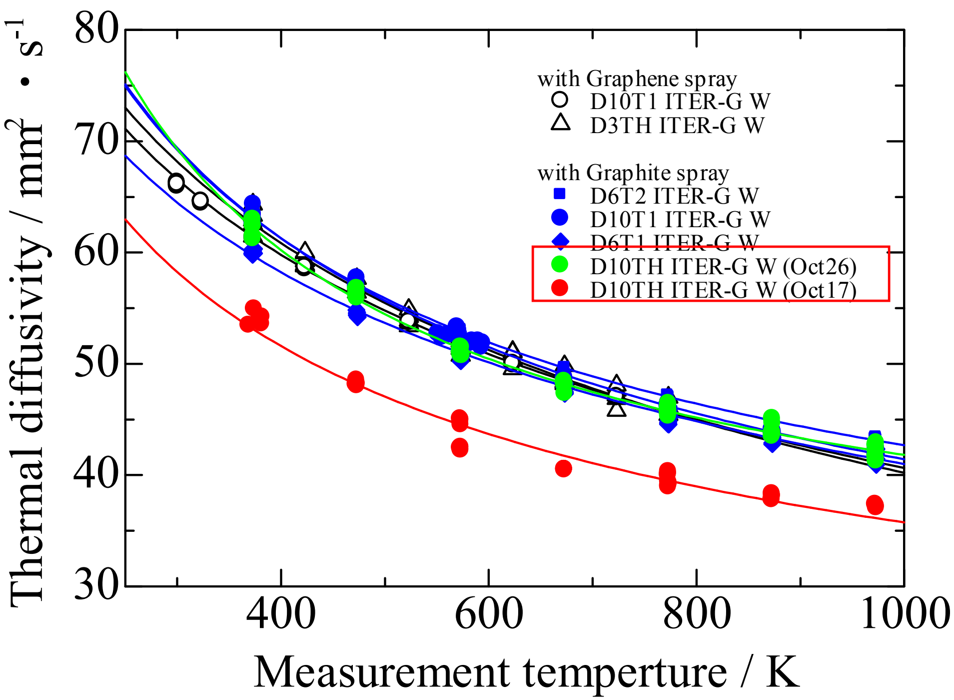
different form Bridgestone Pure-beta β-SiC specimens were measured using NETZSCH LFA-467HT at ORNL.

The results were analyzed with Cowan Model (only one axis thermal diffusion is considered).

>150℃, all specimens showed quite good agreement

SiC specimen does not need to prepare surface coating.

Importance of surface treatment



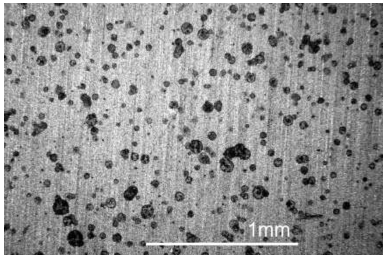
The measurement method was already established. On the other hand, measurement of PHENIX specimens during 2018 was quite limited because of NETZSCH US sold conventional GRAPHITE spray as GRAPHENE spray, and it take a little long time to clarify it.

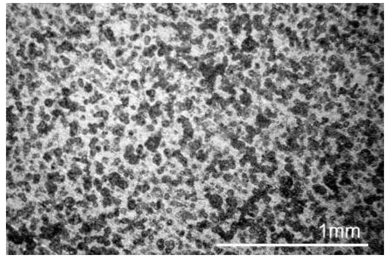
The spray bottle is treat as dangerous object and cannot ship by air, and surface transport by ship takes about 2monthes.

For 1mm thick W specimen, graphite spray is good enough.

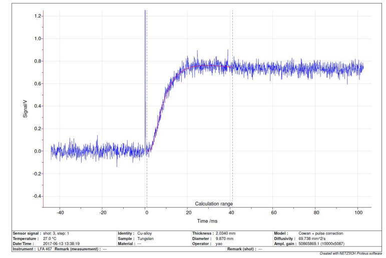
On the other hand, 0.5mm thick specimens sometime showed smaller value and we cannot use graphite spray for unknown specimen.

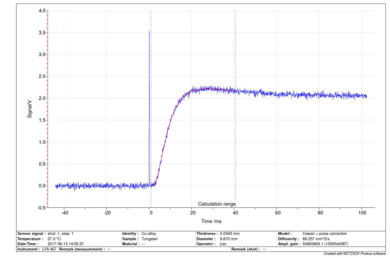
Surface Preparation





Very sparse black coating performed by Graphene nanoplatelets coating agent is required. Density change of the black dots did not affects the obtained thermal diffusivity.



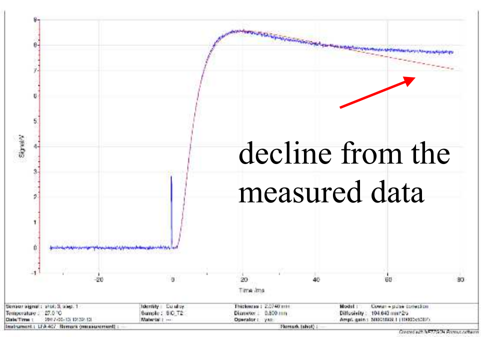


Additional splay gives larger absorptions of flash light and IR radiation that improved the S/N of the profile.

→ But too thick carbon layer cause additional time on the measurement profile.

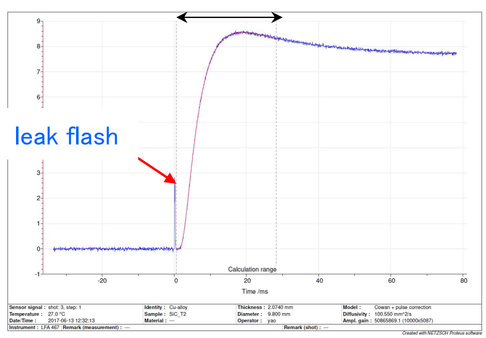
Data Fitting

Fit with full range



TD: 104.6mm^2/s

Fit within $5 \times T_{1/2}$ period



TD: 100.6mm^2/s

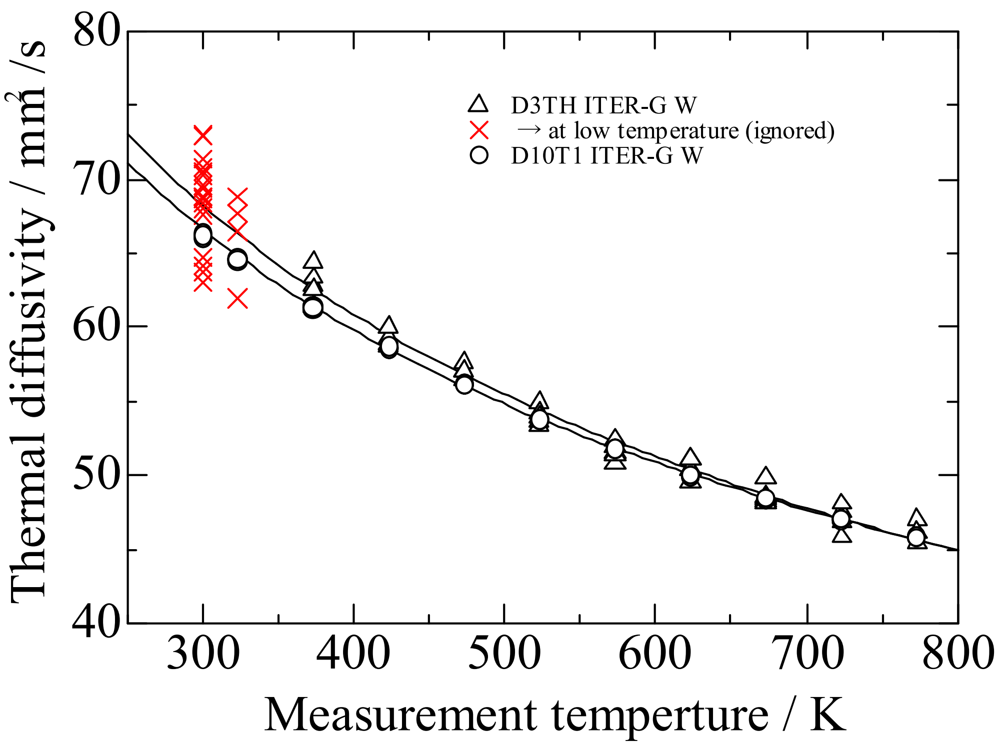
Thermal diffusivity was obtained based on Cowan model fitting + Flash Pulse Collection.

The fitting range was fixed with 5 times of the half time ($T_{1/2}$) and the term during leak flash was avoided at the previous paper. Additional work showed 6-7 × $T_{1/2}$ range gives better matching confirmed by reference specimens.

Validation of measurement for D3TH tungsten specimens with graphene spray using LFA-467

Polished metal surface was covered with brand new graphene nanoplatelet spray.

(Using LFA-467 in Japan (Prof. Kasada))



Extrapolation from data measured at high temperature

D10T1: 66.6mm^2/s

D3TH: 68.2mm^2/s

→ Only 2.3% error

Measurement method of thermal diffusivity for D3TH specimen using LFA-467 is established

M. Akiyoshi, et al., Fusion Engineering and Design, 136 Part A (2018) 513–517